

*Preparation and Characterization of Activated Carbon from Waste of Jengkol Shell (*Pithecellobium jiringa*)*

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Abstract - The purpose of this study was the preparation and characterization of activated carbon from jengkol shell waste. Activated carbon was made through the process of dehydration, carbonization, and activation. The method used for making carbon from jengkol shell was pyrolysis with variations in the pyrolysis temperature of 300°C, 330°C, and 350°C. The activation stage uses a chemical activation method that was using a variety of activated reagents which is HCl, KOH and ZnCl₂ and variations of concentration of activated reagents which is 2N, 4N, and 6N. The activated carbon that was obtained will be tested with the following parameters: water content, ash content, vapor content and bounded carbon content. In this study, the standar used to determine the characteristics of activated carbon was the Indonesian National Standard (SNI 06-3730-1995). The results showed that the carbonization temperature of 300°C and the activated reagent of HCl 4N showed the most optimal result of activated carbon where the water content was 1,72%, the ash content was 0,39%, the vapor content was 10,78%, and the bounded carbon content was 88,83 %. Based on these results it can be concluded that the jengkol shell waste can be used as an activated carbon that meet the Indonesian National Standards (SNI 06-37301995).

Keywords — Activated carbon, jengkol shell, pyrolysis, chemical activation, SNI 06-3730-1995.

I. INTRODUCTION

Industrial development is increasing along with the development of science and technology, so industry is one of the important sectors in the Indonesian economy, one of which is the activated carbon industry[1]. Activated carbon is usually used as an adsorbent in the processing and purification of water and the removal of polluting gases [2,3]. Activated carbon is the most commonly used adsorbent and is proven to be the most effective for removing various organic and inorganic, polar and non-polar compounds and also used in the field of energy storage [4,5].

Activated carbon is a porous material with a content of 85 - 95% carbon produced from carbon-containing materials by heating at high temperatures [6]. Activated carbon is generally produced from woody biomass, agricultural waste and coal through pyrolysis such as wood powder[7], coconut shell, peat [8], rice husk[9], pistachio nut shell[10], grain sorghum [11], jengkol shell[12], lignite and animal bones [13]. The activated

carbon that will be made in this study comes from jengkol shell waste. Jengkol shells have been classified as organic waste scattered on traditional markets and do not provide economic value. Therefore the processing of jengkol shells as activated carbon is one way to increase the economic value of the waste so that it is not wasted [12].

In general, the preparation of activated carbon consists of 3 stages, which is dehydration, carbonization and activation. Dehydration is the process of removing water in raw materials. Carbonization is the burning of raw materials using limited air with air temperatures between 300°C - 900°C according to the hardness of the raw material used [14]. This combustion process causes the decomposition of organic compounds that make up the structure of material in the form of cellulose, hemicellulose and lignin, and forms water vapor, methanol, acetic acid vapor and hydrocarbons. With the carbonization process, the flying substances contained in briquettes are lowered as low as possible, so that the final product is not gray

and smoky[15]. Solid material left behind after the carbonization process is carbon in the form of charcoal with a narrow specific surface [14]. The next step is activation. Activation is an important process in making activated carbon. Activations aims to enlarge and remove impurities in the pores of activated carbon by breaking the hydrocarbon bonds. Activation consists of two ways, which is physical activations and chemical activation. On physical activation, carbon was activated at a high enough temperature using steam or gases such as carbon dioxide as an activated reagent[16]. Chemical activation is the process of breaking the carbon chain from organic compounds by using chemicals such as phosphoric acid (H_3PO_4), calcium chloride ($CaCl_2$), sodium chloride ($NaCl$), calcium hydroxide $Ca(OH)_2$, hydrochloric acid (HCl), magnesium dichloride ($MgCl_2$), zink chloride ($ZnCl_2$), sodium hydroxide ($NaOH$), potassium hydroxide (KOH), nitric acid (HNO_3 etc.[14].

The quality requirements for activated carbon according to the Indonesian National Standard (SNI 06-3730-1995) are shown in table 1 [17].

Tabel 1. active carbon quality requirements based on Indonesian national standards (SNI 06-3730-1995)

Type of requirements	parameter
Substances Evaporates	Max. 25 %
Moisture content	Max. 15%
Ash content	Max. 10%
Bounded Carbon content	Min. 65%

The purpose of this research was to determine the optimal of pyrolysis temperature, determine the optimal of activated reagents and the optimal of concentration of activated reagents to obtain high quality, economical and environmentally friendly activated carbon.

II. RESEARCH METHOD

A. Preparation Activated Carbon of Jengkol Shell

Jengkol shells were obtained from Lubuk Alung Market, Padang Pariaman Regency. Jengkol shells were dried in sunlight to reduce the water content to produce good activated carbon.

B. Carbonization and Activation of Jengkol Shell

The carbonization stage, 500 grams of jengkol shell were pyrolysis in the furnace at a temperature variation of 300 °C, 330°C, and 350°C for 1 hour. The carbon resulting from the carbonization stage were smoothed using mortar and pestle, then filtered using a 100 mesh or 150 µm sieve.

The activation stage was done by immersing 5 grams of carbon from the pyrolysis using different activated reagents (HCl , KOH , and $ZnCl_2$) each with a concentration of 4N and the volume was 25 mL for 24 hours. Then filtered using filter paper and washed with aquades to neutral pH, oven at a temperature of 110°C for 1 hours. After obtaining the optimal activated reagent, the concentration varied from 2N, 4N and 6N to get the optimal concentration of activated reagent.

C. Characteristics of Activated Carbon

The obtained of activated carbon was tested with the following parameters:

1) Water Content Analysis

One gram of activated carbon was put into a dried porcelain crucible which has been dried, then put in the oven at 105°C for 1 hour, then cooled in the desiccator and weighed. Water content can be calculated by the following equation:

$$\text{Water content} = \frac{a-b}{a} \times 100\%$$

Where:

a = initial activated charcoal weight (gram)

b = weight of activated charcoal after drying (gram)

2) Ash Content Analysis

One gram of activated carbon was put into porcelain crucible. Then in the furnace at a temperature of 500°C for 1,5 hours. If all the carbon has become ash, cool it in the desiccator and then weight it until its fixed weight was obtained.

$$\text{Ash content} = \frac{\text{weight ash}}{\text{weight of sample}} \times 100\%$$

3) Vapor Content Analysis

Activated carbon was heated to a temperature of 310°C in the furnace. After the temperature was reached, the carbon was allowed to cool in the furnace by not relating to outside air. After cool was put into the desiccator and weighed. Vapor content was calculated with this formula :

$$\text{Vapor content} = \frac{a-b}{b} \times 100\%$$

Where:

a = initial activated carbon weight (gram)

b = weight of activated carbon after heated (gram)

4) Bounded Carbon Content Analysis

The bounded carbon content of activated carbon was obtained from the results of the reduction of the part that is lost in heating 310°C (vapor content) and ash content.

$$\text{Bounded carbon} = 100\% - (A+B)$$

Where :

A = ash content (%)

B = vapor content (%)

III. RESULTS AND DISCUSSION

A. Preparation Activated Carbon of Jengkol Shell

Activated carbon was carried out through two stages: carbonization and activation stage. Jengkol shell samples that have been prepared are carried out a washing process that aims to remove impurities found in the sample. After washing, the sample was dried first under sunlight to dry before the pyrolysis process. This is so that the furnace does not smoke too much during the pyrolysis process.

B. Carbonization and Activation of Jengkol Shell

1) Variation of Carbonization Temperature

The carbonization stage is the step of changing jengkol shells into charcoal (carbon). Jengkol shell samples were burned in the furnace with temperature variations of 300°C (CA), 330°C (CB), and 350°C (CC) each 1 hour, then tested: water content, ash content, vapor content, and bounded carbon content. CA is a symbol of jengkol shell pyrolysis at a temperature of 300°C, CB is a symbol of jengkol shell pyrolysis at a temperature of 330°C while CC is a symbol of jengkol shell pyrolysis at a temperature of 350°C.

a) Water Content Analysis

Water content testing was carried out to determine the remaining water content on carbon.

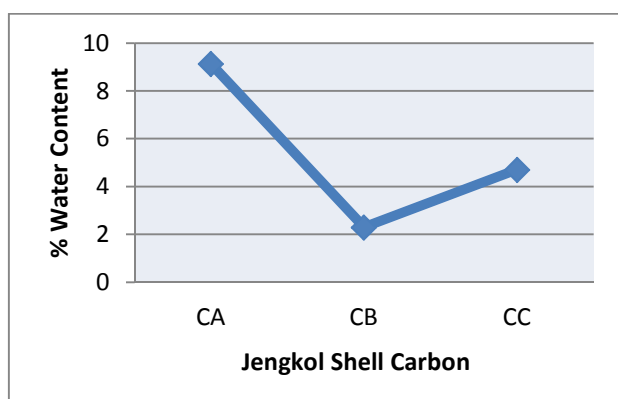


Fig. 3.1 Water content analysis of carbon from pyrolysis process of jengkol shells

Figure 3.1 shows the results of the water content of carbon from pyrolysis process at temperature variations of 300

°C, 330 °C and 350 °C. Based on the graph it is known the water content at the carbonization temperature of 300°C was higher than 330 °C and 350 °C which is 9.13%, while at temperature of 330 °C the water content was found at 2.29% and at 350 °C was 4.70%. The lower the carbonization temperature, the higher the water content. The above water content values are still included in the SNI range. According to SNI the value of water content for activated carbon is at least 15%.

b) Ash Content Analysis

Analysis of ash content was useful for determining the content of metal oxides that are still present in carbon after going through the carbonization process.

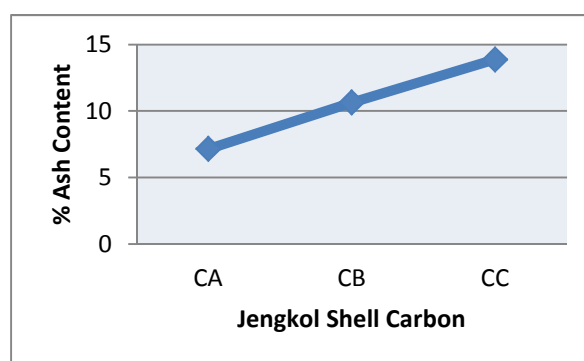


Fig. 3.2 Ash content analysis of carbon from pyrolysis process of jengkol shells

Figure 3.2 shows that at the carbonization temperature 300°C has the lowest ash content of 7.157%, then at the carbonization temperature 330°C and 350°C the ash content increases, at temperature 330° was 10.64% and at 350°C was 13.875%. The higher the carbonization temperature, the greater the ash content produced. If the temperature is too high, the carbon will turn to ash [18].

c) Vapor Content Analysis

Analysis of vapor content serves to determine the amount of substances or compounds that have not evaporated after carbonization process.

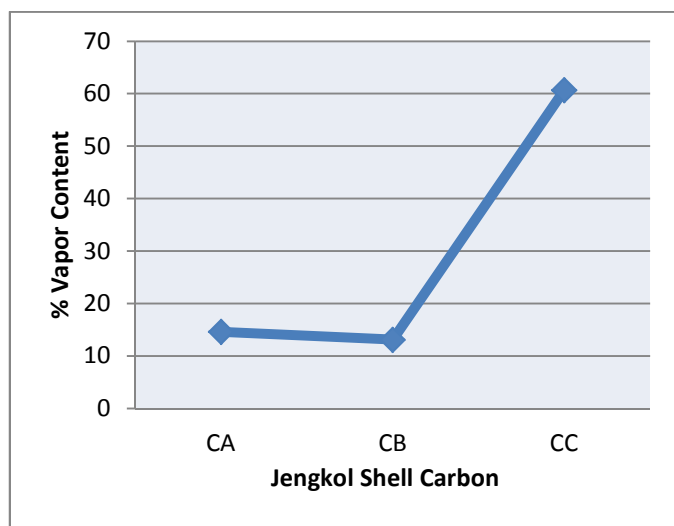


Fig.3.3 Vapor content analysis of carbon from pyrolysis process of jengkol shells

Figure 3.3 shows that at temperature of 300 °C the vapor content was found at 14,46%, at 330°C was 13,09% and at 350°C was 60,67%. Based on the graph it can be concluded that the higher the carbonization temperature, the more volatile substances that evaporate.

d) Bounded Carbon Content Analysis

The testing of bounded carbon content aims to determine the carbon content after the carbonization process. Bound carbon content was calculated from the values of vapor content and ash content.

Figure 3.4 shows the test results of bounded carbon content to variations in carbonization temperature of 300 °C, 330 °C, and 350 °C. At temperature of 300°C the bounded carbon content was found at 78,28%, at 330°C was 76,27% and at 350°C was 25,45%. At a temperature of 300 °C has the highest bounded carbon content. The higher the carbonization temperature, the lower the bounded carbon content produced.

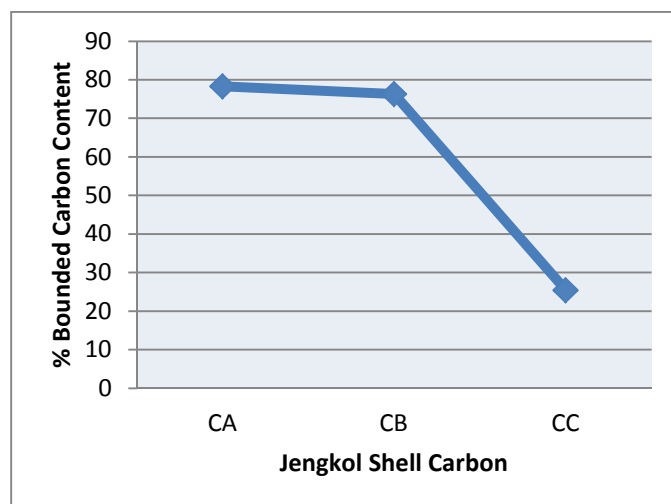


Fig. 3.4 bounded carbon content analysis of carbon from pyrolysis process of jengkol shells

Based on the test of water content, vapor content, ash content, and bounded carbon content to the variations in carbonization temperature of 300 °C, 330 °C, and 350 °C it can be concluded that the optimal carbonization temperature of jengkol shells was 300°C (CA) with a water content of 9,13%, ash content of 7,157%, vapor content of 14,56% and bounded carbon content of 78,28%.

2) Variation of Activated Reagents

The next stage was the activation stage, which is the change in carbon into activated carbon. The carbon from the pyrolysis was soaked with a variety of activated reagents, which is KOH, ZnCl₂, and HCl for 24 hours with a concentration of 4N each. The purpose of adding activated reagents is as an activator to break the hydrocarbon bonds. The immersion results were washed with aquades to neutral pH which aims to clean the remaining activating solution which is still left behind. The activation results are in the oven at 110°C for 1 hours which aims to vaporize the water content. Then several tests were carried out, which is water content, ash content, vapor content and bounded carbon content.

a) Analysis of water content

Water content analysis was carried out to determine the water content after going through the process of activation.

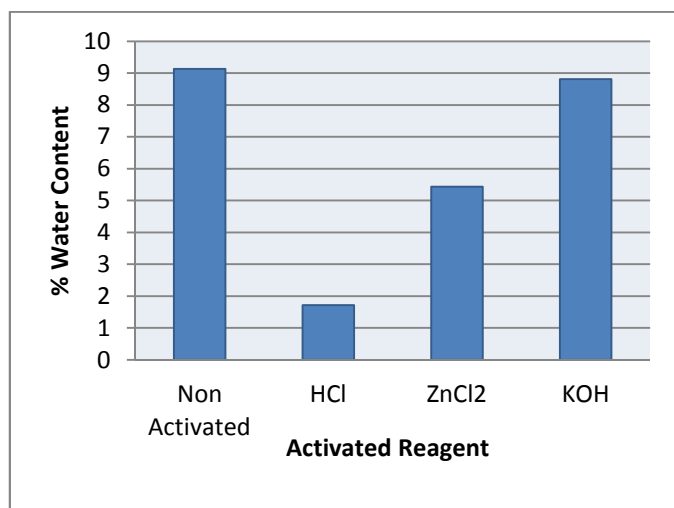


Fig. 3.5 Water content analysis of jengkol shell CA at various activated reagent at 4N concentration

Figure 3.5 shows a significant graphical change between carbon without activation and after activation. Carbon without activation has the highest water content of 9,13% and activated carbon with HCl activator solution has the lowest water content of 1,72%. According to SNI the value of water content of carbon without activation and after activation was allowed a maximum of 15%.

b) Ash Content Analysis

The ash content test aims to determine the metal oxide content that is still present in activated carbon after going through the process of activations.

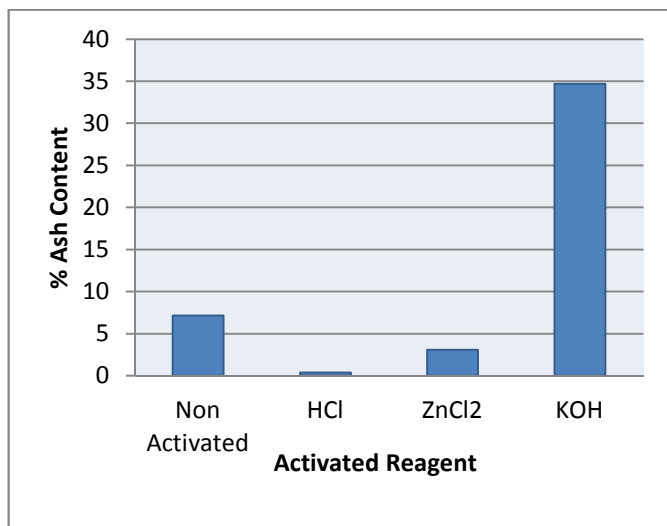


Fig 3.6 Ash content analysis of jengkol shell CA at various activated reagent at 4N concentration

Figure 3.6 shows significant graphical changes after activation with HCl activated reagents. Carbon without activation has an ash content of 7,157% while after activation with HCl 4N solution the ash content becomes 0,39%. In the graph it can be seen that the ash content of activated carbon with KOH activating reagents is higher at 34,69%. It can be concluded that the KOH solution was not suitable as a activated reagent the jengkol shell and activated reagent which is good for activating jengkol shells, namely HCl solution because it has a low ash content.

c) Vapor Content Analysis

Analysis of vapor content serves to determine the amount of substance or compounds that have not evaporated after activation process.

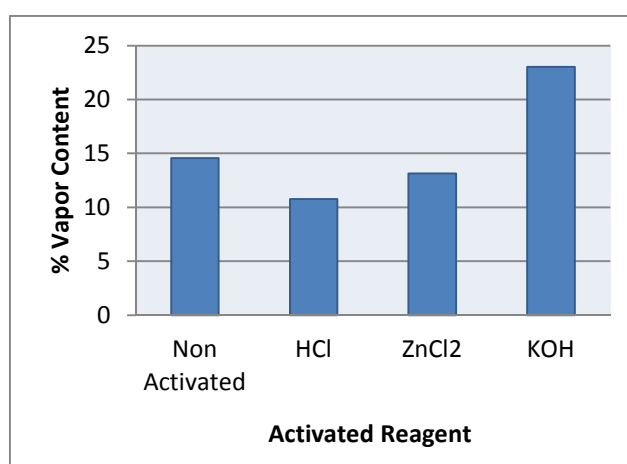


Fig 3.7 Vapor content analysis of jengkol shell CA at various activated reagent at 4N concentration

Figure 3.7 shows the results of the test of carbon vapor content without activation and after activation. The vapor content with the KOH activated reagent was the highest, which is 23,04%. It can be concluded that the KOH solution was not suitable as a activated reagent for jengkol shell and a good activated reagent for the activation of jengkol shells namely HCl solution because it has a low vapor content of 10,78%.

d) Analysis of Bounded Carbon Content

Analysis of bounded carbon content serves to determine the carbon content after going through the activation process.

Figure 3.8 shows the results of bounded carbon content tests to variations of activated reagents. Activated carbon with the HCl activated reagent has the highest bounded carbon content, which is 88,83%, while KOH has the lowest bounded carbon content of 42,27% and was not included in the SNI range. Based on SNI 06-3730-1995 the bounded carbon

content to activated carbon was at least 65%. It can be concluded that KOH cannot be used as an activator solution for activation of activated carbon from jengkol shells.

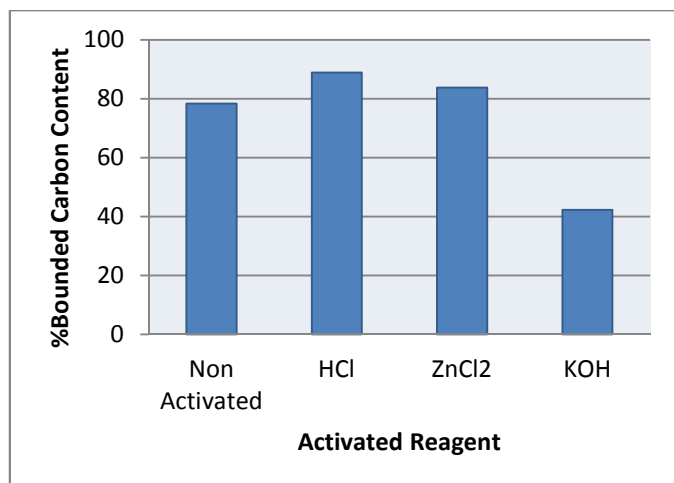


Fig 3.8 Bounded carbon content analysis of jengkol shell CA at various activated reagent at 4N concentration

Based on the test of water content, vapor content, ash content, and bounded carbon content to variations of activated reagents it can be concluded that the optimal activated reagent for activation of activated carbon from jengkol shell was HCl with a water content 1,72%, ash content 0,39%, vapor content 1078% and bounded carbon content 88,83%.

3) Variations of Activated Reagent Concentration

After obtaining the optimal activated reagent, which is HCl, then the concentration varied from 2N, 4N, and 6N

a) Water Content Analysis

Water content testing was carried out to determine the water content remaining on activated carbon after going through activation process based on variations in the concentration of activated reagents.

Figure 3.9 shows the water content of activated carbon with variations in the concentration of HCl solution. Activated carbon with activated reagent HCl 4N has the lowest water content of 1,72% and activated carbon with 6N HCl activator solution has the highest water content of 2,78%. At a concentration of 4N the activated carbon works optimally because it produces the lowest water content.

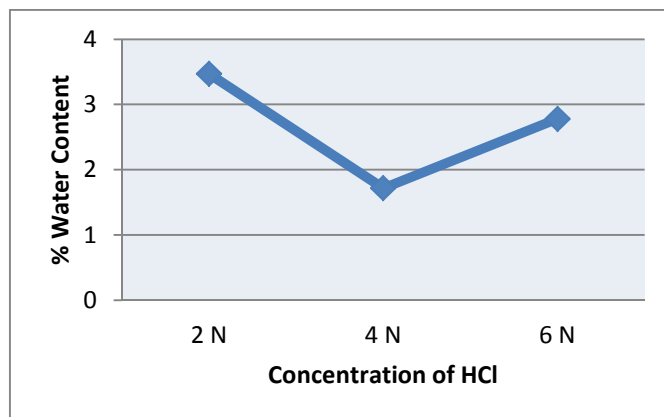


Fig. 3.9 Water content analysis of jengkol shell CA activated carbon with HCl concentration at various concentration

b) Ash Content Analysis

Analysis of ash content was useful for determining the content of metal oxides that are still present in activated carbon after going through the activation process based on variations in the concentrations of activated reagents.

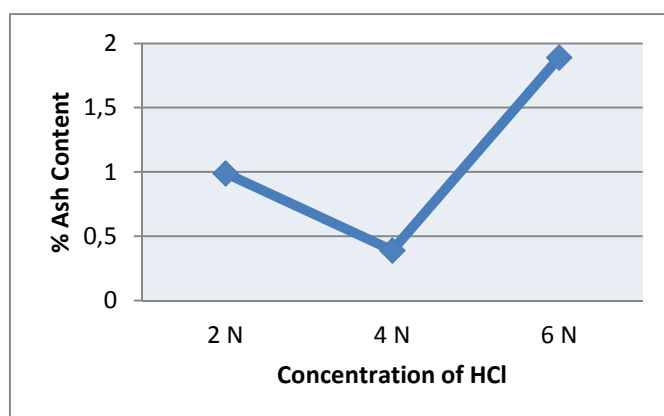


Fig. 3.10 Ash content analysis of jengkol shell CA activated carbon with HCl concentration at various concentration

Figure 3.10 shows activated carbon with activated reagent HCl 4N has the lowest ash content of 0,39% and activated carbon with activator solutions of 6N HCl having the highest ash content of 1,89. At the concentration of HCl 4N the activated carbon works optimally because it produces the lowest ash content. The value of ash content increases with increasing concentration of activator substances, because too many activators will cause clogging of the pores of activated carbon causing high ash content [19].

c) Vapor Content Analysis

Analysis of vapor content serves to determine the amount of substance or compounds that have not evaporated after activation process based on variations in the concentration of activated reagents.

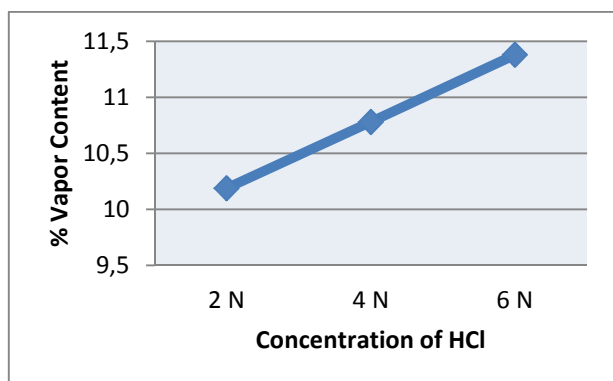


Fig.3.11 Vapor content analysis of jengkol shell CA activated carbon with HCl concentration at various concentration

Figure 3.11 shows the vapor content of activated carbon with variations of HCl solution with 2N, 4N and 6N. At concentration HCl 2N the vapor content of activated carbon was found 10,19%, at HCl 4N was 10,78% and at HCl 6N was 11,38%. Activated carbon with 2N HCl activated reagent has the lowest vapor content which is 10,19%. The higher the concentration, the higher the vapor concentration. Vapor content values of various activating reagent concentrations are all included in the SNI range. According to SNI the maximum value of vapor content was 25%.

d) Analysis of Bound Carbon Content

Analysis of bounded carbon content serves to determine the carbon content after going through the activation process based on variations in the concentration of activated reagents.

Figure 3.12 shows the bounded carbon content of activated carbon by variations in the concentration of HCl solution. The activated carbon with the activated reagent of 4N HCl has the highest bounded carbon content of 88.83%. while the activated carbon with 2N HCl activated reagent has a 88,82% bounded carbon content. Whereas the activated carbon with the activated reagent HCl 6N had the lowest bounded carbon content of 86,73%. The value of bounded carbon content can be calculated from the values of vapor content and ash content. The smaller the value of the vapor content and the ash content of an activated carbon, the higher the value of the bounded carbon, this indicates the better quality of the activated carbon.

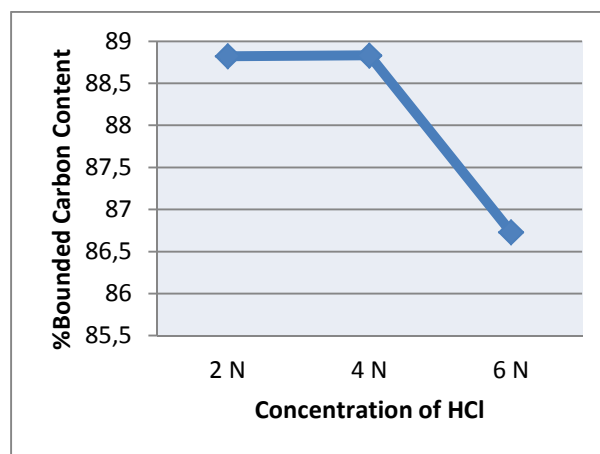


Fig.3.12 Bounded carbon content analysis of jengkol shell CA activated carbon with HCl concentration at various concentration

According to the test of water content, vapor content, ash content, and bounded carbon content to variations in the concentration of activated reagents it can be concluded that the best concentration of activated reagents for activation of jengkol shells was HCl 4N with a water content of 1,72%, ash content 0,39%, vapor content 10,78% and bounded carbon content 88,83%.

IV. CONCLUSION

Activated carbon can be made from jengkol shell biomass waste. Optimal pyrolysis temperature for carbonization of jengkol shell was 300°C for 1 hour and the optimal activated reagent for activation of activated carbon from jengkol shell was HCl 4N solution. Activated carbon from jengkol shell meets the quality requirements of activated carbon based on SNI 06-3730-1995 where the water content was 1,72%, ash content was 0,39%, vapor content was 10,78% and bounded carbon content was 88,83 %.

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